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INVESTIGATION OF CHANGES IN THE FINE INTRAGRANULAR STRUCTURE OF A COMPLEX COBALT-BASE ALLOY (KhCNKhM) DURING PLASTIC DEFORMATION AND ANNEALING

- USSR -

by Yu. A. Skakov and Ya. S. Umanskiy

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INVESTIGATION OF CHANGES IN THE FINE INTRAGRANULAR STRUCTURE OF A COMPLEX COBALT-BASE ALLOY (KhONKhM) DURING PLASTIC DEFORMATION AND ANNEALING

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[Following is a translation of an article by Yu. A. Skakov and Ya. S. Umanskiy in the Russian-language periodical Izvestiya Vysshikh Uchebnykh Zavedeniy, Chernaya Metallurgiya (News of the Higher Educational Institutions Ferrous Metallurgy), Moscow, No 5, May 1960, pages 150-158.]

The alloy KhONKhM [1,2] is exceptionally strongly strengthened during plastic deformation and, furthermore, experiences substantial additional strengthening during low-temperature annealing. An explanation of the character of the structural changes relative to strengthening and weakening was the basic aim of the present study.

The composition of the alloy studied was as follows: 0.08% C; 36.4% Co; 20.1% Cr; 15.25% Ni; 7.05% Mo; 16.3% Fe; 0.40% Si; 1.82% Mn. Specimens of the alloy were subjected to cold forging (10, 30, 50 and 70% of compression) after hardening at 1150° in water. Annealing was done at temperatures from 100 to 900° during a period of four hours. At 500° the duration of annealing was changed from 2 to 100 hours; at 700°, to 18 hours. The microhardness of all specimens was measured. (The experimental work was carried out with the participation of A. V. Sharshatkin and students of MIS [Moskovskiy Institut Stali -- Moscow Steel Institute] L. K. Kostin, M. M. Arengol'd. Considerable assistance in the choice of objectives and their preparation for study was shown on the part of D. I. Gabrielyan and other workers of IPS TsNIIChM [Institute Proizvodstva Stali Tsentral'nyy nauchno-issledvotel'skiy institut chernoy metallurgiya -- Institute of Steel Production Central Scientific Research Institute of Ferrous Metallurgy] in particular V. A. Sol'ts.)

The method of single-stepped, carbon (ugol'nykh) prints with fixed particles in the case of heterogeneous composition was used for electron-microscope research.

Electron diffraction research was carried out "in the reflection" of etched microsections and "in the gap" of varnished prints with fixed particles of the second phase and also of the films of the alloys obtained by the electrolytic thinning of massive specimens [3].

Two series of radiographs with linear focusing (111) and (220) in Cr-radiation were taken to determine the size of the blocks and the micro-deformation of the crystal lattice of the solid solution.

The size of the blocks and the micro-deformations were calculated by the method of harmonic analysis of the shape of the curve of true or physical widening [4,5]. A hardened specimen was taken as a standard. The decomposition in a number of standard and widened photometric curves were made with the aid of patterns during the division of the interval of decomposition into 40 parts and into 80 parts (in order to obtain intermediate values of the coefficients [6].

Results of the Experiments

1. The measurement of electrical resistance in the process of heating and working the preliminarily deformed specimens showed that a process develops at heating temperatures of about 400-450° which increases electrical resistance. (Measurement was made by the compensation method; the speed of heating and cooling was 2-3° per minute.) At heating temperatures above 550-600°, a process takes place which leads to a decline in the electrical resistance. Above 900° the reverse process proceeds intensely. These results coincide with the results of studies [1,2].

The nature of the transformation at annealing temperatures greater than 550-600° is clear: down to 850-900° a process of separation from the solid solution takes place, then a process of reverse dissolution.

The effect of increasing the electrical resistance during annealing below 500° is explained in a different way: in study [1] the action of the phase cold hardening is assumed owing to the division of the second phase; in study [2] this fact belongs to the so-called "x-anomaly" (considered by the authors as a special case of regulation) that is analogous to the term "K-condition" adopted in German and other literature.

As is clear in Figure 1, additional strength during annealing occurs in the temperature range 300-500°, i.e., in that range in which the anomalous change in electrical resistance was observed. Annealing at temperatures of 600-650° and above leads to a decline in hardness. Maximum additional strengthening was observed after

annealing at 500° for four hours. With an increase in annealing time, this strengthening is removed; however, the hardness practically does not change during up to 100 hours of further soaking.

2. Quite substantial crushing of the blocks was noted in analysing the widening of line (220) after deformation at 30% (Figure 2). The crushing of the blocks is observed even during further increases in the degree of deformation. (During deformation 10% of the effect of widening of the lines is not noticed, i.e., the dimensions of the blocks remain greater than 0.1 μ .)

An interesting peculiarity of changes in the mosaic structure as a result of plastic deformation is the anisotropy of the granulation of the blocks. The difference in dimensions of the blocks calculated from reflections (220) and (111) for all three degrees of deformation (30%, 50% and 70%) is found in the limits of the computational error of the method. It can be assumed that in actuality the dissimilarity in dimensions is greater, but this dissimilarity conceals the conditions of exposing the radiograph in which neutralization inevitably occurs. The presence of texture diminishes the degree of this neutralization.

From the electron diffraction data it follows that the texture of forging is (110) [111] and (110) [111]. It can be assumed that during the plastic deformation of the block it acquires an elongated form in the direction [111], established along the direction of forging.

Increasing the degree of deformation to 70% leads to great crushing of the blocks (up to $\sim 10^{-6}$ cm) and simultaneously to a decline in the diversity of block dimensions in various directions.

The conclusion on the significant size of the blocks in direction [111] after deformation at 30-50% is supported by the analysis of changes in the intensity of line [111] following hardening, deformation and strength tempering and, in addition, after cold working the surface of these same specimens with emery paper.

	Intensity (111), %
Annealing	25
Annealing and Cold Working of the Surface	87
Cold Forging (50% of Compression)	29
Cold Forging and Annealing at 500°	40
Cold Forging and Cold Working of the Surface	100

If the increase in intensity for deformed and annealed specimens after cold working of the surface are in some measure the result of a change in the texture, the increase in intensity of the deformed specimen after strength annealing can be explained only

by the decrease in the extinction effect. (The determination of the integral intensity was carried out on a URS-501 apparatus.) For the present, the role of packing defects remains unclear in the effects of changes in the breadth and intensity of line (111).

3. The maximum values of micro-deformation (ε_{max}), calculated by reflections (220) and (111) for all practical purposes coincide. However, the micro-deformation of the lattice in direction [110] is localized in lesser volumes than in direction [111] (Figure 3). (The size of the area of localization of micro-deformations can be judged by the spacing of L (in angstroms) which corresponds to the maximum of curve $\varepsilon(L)$.)

With an increase in the degree of compression to 50%, the micro-deformations increase sharply and attain values of $4 \cdot 10^{-3}$, which clearly is the limit.

If it is assumed that the quantity determined by us is connected only with microstrains, a rough evaluation of these strains provides a quite high number -- about 100 kg/mm^2. It is clear, however, that the micro-deformations of the crystal lattice includes not only elastic distortions but also distortions connected with concentrated heterogeneity.

It is interesting to compare our data with the results of a study by Gadfield [6] on the micro-deformations of a lattice during cold steel forging which is also considerably strengthened. The maximum size of the micro-deformations in this steel with 30-70% of deformation does not change and consists in all of $2.5 \cdot 10^{-3}$, i.e., 1.5 - 2 times less than in the alloy KhONKhM. In alloy KhONKhM which contains elements differing considerably in atomic radii, one would expect a great effect of rising diffusion, basically as a result of the difference of the atomic radii of molybdenum ($D_{at} = 2.80$ Å) and other components ($D_{at\ Co} = 2.5$ Å); $D_{at\ Cr} = 2.57$ Å; $D_{at\ Ni} = 2.49$ Å; $D_{at\ Fe} = 2.52$ Å).

The characteristic relief of the traces of deformation observed in photomicrographs of pickled microsections testify to the presence of sharp heterogeneity in the alloy (See Figure 4,a).

In the electron diffraction study, a second system of lines corresponding to $\frac{d}{n} \approx 2.16$ and ≈ 1.97 Å is disclosed in the gap of a fine film of the specimen. The lines have exactly the same arrangement of textural maximums as the lines of the matrix (111) and (200). The appearance of these lines could be explained by the presence of segregations of molybdenum and carbon. However, these lines are connected with disturbances in the arrangement of close-packed surfaces and the appearance of portions with an hexagonal structure.

4. Annealing at 500° (maximum hardness) leads to the growth of lattice micro-deformations up to that limiting value which was noted in strongly deformed specimens ($\approx 4 \cdot 10^{-3}$). After annealing at a temperature of 700° (weakening takes place), micro-deformations of the lattice decline very sharply, reaching approximately the very same magnitude $\varepsilon_{max} \approx 2 \cdot 10^{-3}$ both for the specimen preliminarily deformed at 30% as well as for the specimen deformed at 70% (Figure 5).

Changes in the size of the blocks after annealing at 500° are comparatively small. After annealing at 700°, the sizes of the blocks increase, accompanied by a sharp weakening (Figure 5). Additional lines disappear in the electron diffraction picture of fine film.

X-ray and electron optical data testify to the fact that as a result of annealing at 500° considerable changes take place within the solid solution. These changes consist in the strengthening of the concentration of heterogeneity as a consequence of the diffusion of molybdenum and carbon in the elastically elongated area of the crystal lattice of the solid solution.

After annealing at 500° electron optical separation of the carbide phase is not revealed. The relief of the pickled microsection becomes clearer. Portions of the microrelief of the magnitude 0.01 - 0.1 μ (Figure 4, b) are easily seen within the band of deformation. This increase in contrast and differentiation of the microrelief can be linked with the increase in the size of the micro-deformations and with a decrease in the size of the area of their localization (Figure 6).

The actual separation of the carbide phase observed electron-optically after annealing at 700° (Figure 4,c) leads to a substantial reduction in the micro-deformation of the lattice. Clearly the portions of the solid solution enriched by molybdenum, carbon and chromium stand apart and constitute the separation of the carbide phase. It is clear from the photomicrographs in Figure 4 that the distribution, sizes and form of the particles of the carbide phase succeed the distribution, sizes and form of micro-heterogeneity which arises during deformation and low-temperature annealing. The sizes of the separations after annealing at 700° following compression at 30% are far less (≈ 100 Å) than following compression at 70% (several hundred angstroms and up to 1,000).

5. Portions of the solid solution containing an increased quantity of molybdenum and chromium can be considered the first product of disintegration. Annealing at a temperature of 700° after heavy deformation in electron diffraction pictures "in reflection" provide a system of lines which coincide closely with x-ray data for carbides of the Co_3Mo_3C type. At the same time, the electron diffraction pictures in the reflection of specimens annealed at

700° after less deformation (for example 30%) and the electron diffraction pictures in the gap of the print with fixed particles for the specimen deformed at 70% and annealed a long time at 700° (18 hours) or at 800° provides a system of lines which correspond better to carbide of the $Cr_{23}C_6$ type. Electron diffraction pictures in the gap of the print with fixed particles of the specimen annealed at 700° for four hours apparently provides two systems of lines, one of which is characteristic for carbide of the Co_3Mo_3C type and the other for carbide of the $Cr_{23}C_6$ type. The conclusion follows from a comparison of these data that the formation of a carbide of the Co_3Mo_3C type depends on the fact that the basis of the alloy is cobalt and as a result of the rising diffusion in the field of strain, an area enriched by molybdenum and carbon appears. It is understandable, therefore, that the formation of this phase proceeds more preferably with a large degree of deformation. Carbide of the $Cr_{23}C_6$ type is clearly the equilibrium phase of disintegration. Its formation is connected with an increase in the chromium and carbon content. The lattice period of this carbide ($a \approx 10.8$ Å) according to our data is somewhat greater than for $Cr_{23}C_6$ ($a \approx 10.62$ Å) which is explained by the presence of a significant quantity of molybdenum in its composition.

Conclusion

Strengthening of an alloy during cold plastic deformation and additional strengthening during heating to 500° are dependent on the very same factors: the crushing of the mosaic blocks (up to $\approx 10^{-6}$ cm) and great micro-deformations of the crystal lattice of the solid solution (up to $\approx 4 \cdot 10^{-3}$). However, the significance of these strengthening factors during deformation and during strength annealing are different. With an increase in the degree of compression from 50 to 70% additional strengthening during low-temperature annealing is linked with further crushing of the blocks. Additional strengthening during low-temperature annealing of a heavily deformed alloy is linked above all with a change in the character of the distribution of the micro-deformations and their increase.

Micro-deformations of the crystal lattice are dependent principally on the fine concentration of heterogeneity within the solid solution. Differentiation of the components (above all molybdenum and carbon) takes place in areas with dimensions on the order of 10^{-6} cm. An increase in the degree of compression and an increase in the length or temperature of annealing leads to an enlargement of the areas of localization of micro-deformations of the crystal lattice of the solid solution (or to the enlargement of portions of the solid solution with an increased concentration of Mo and C). Such a structure proceeds the separation of the carbide phase and corresponds to maximum hardness.

Separation of the carbide phase is accompanied by the removal of micro-deformations of the crystal lattice and the sharp enlargement of the blocks of the mosaic structure which leads to weakening.

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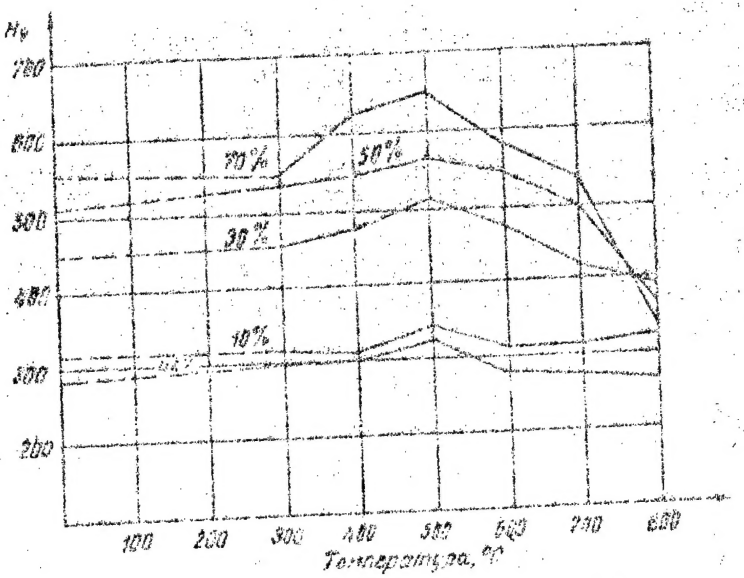
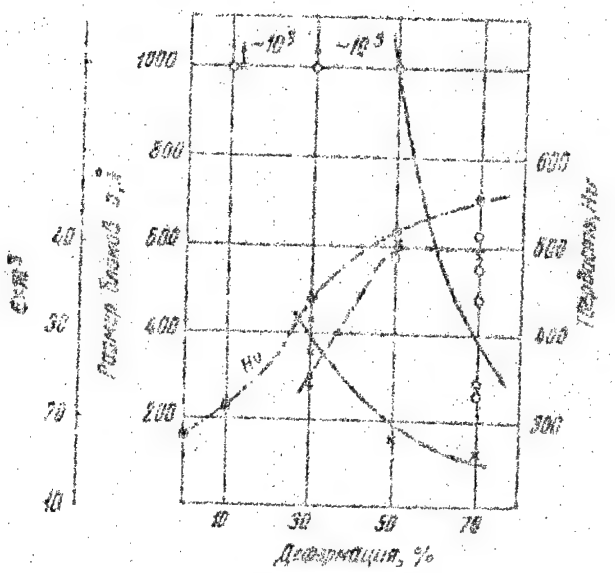
END

FIGURE APPENDIX



Temperature, °C

Figure 1. The dependence of micro-hardness on the annealing temperature (length of annealing, 4 hours)



Size of Blocks D, $\overset{0}{A}$ Hardness, H_v

Deformation, %

Figure 2. The dependence of hardness (- - -), sizes of the blocks (————) and maximum relative micro-deformation of the lattice (------) on the degree of compression: O -- calculation from reflections (111); X -- calculations from reflections (220)

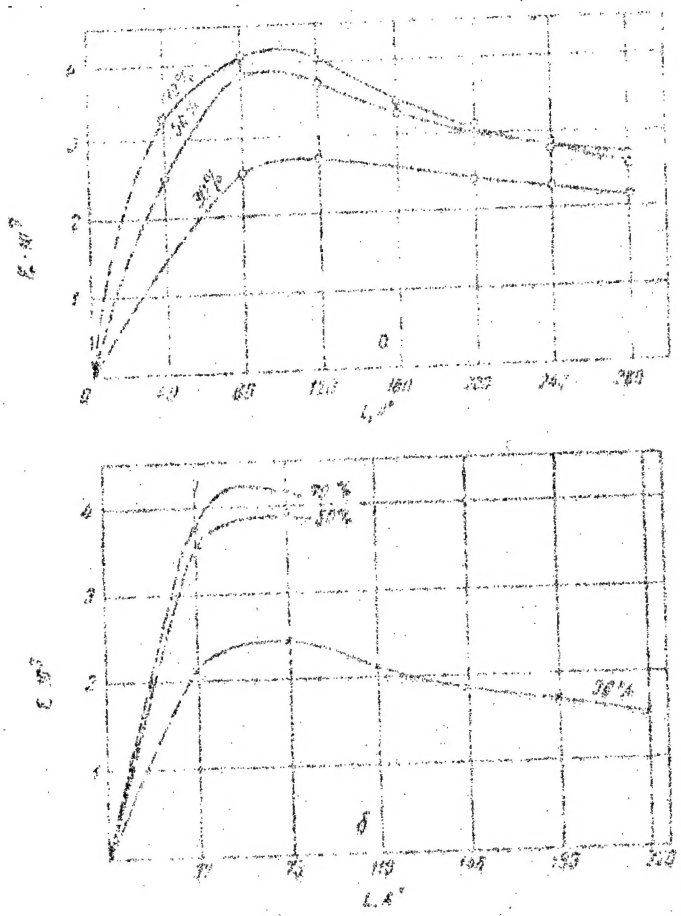


Figure 3. The distribution of relative micro-deformations in a crystal lattice. a -- along direction (111); b -- along direction (110)

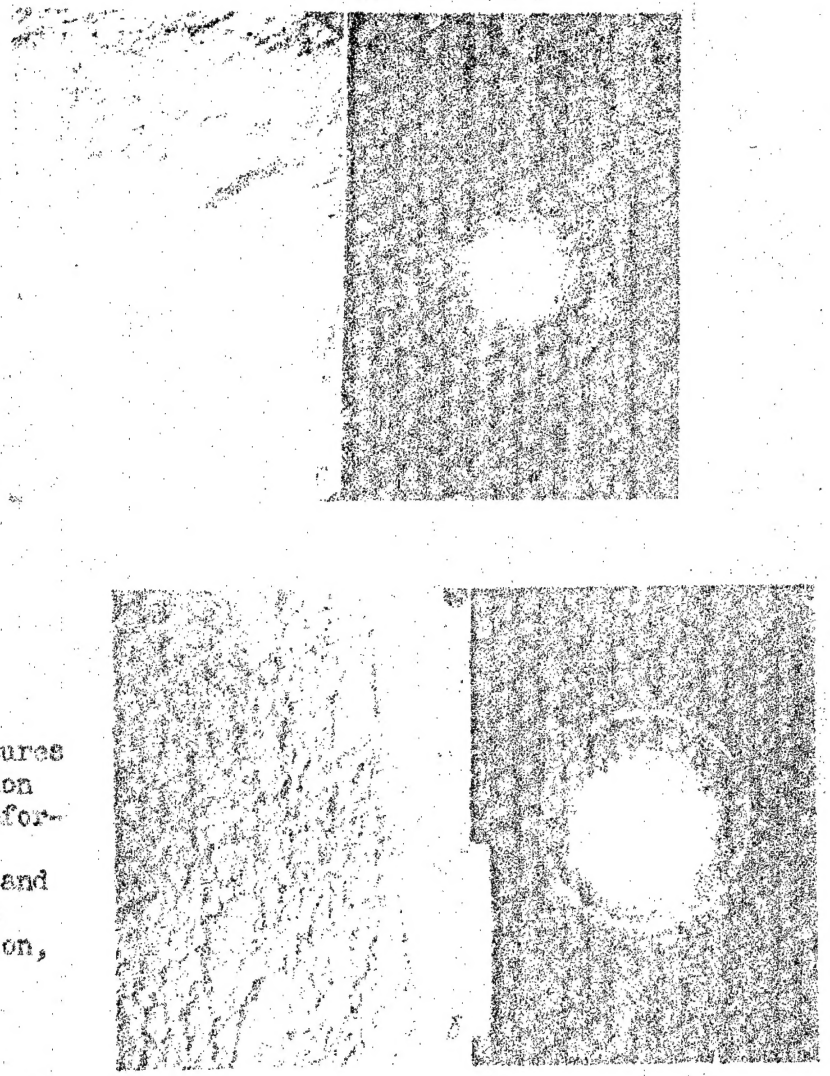
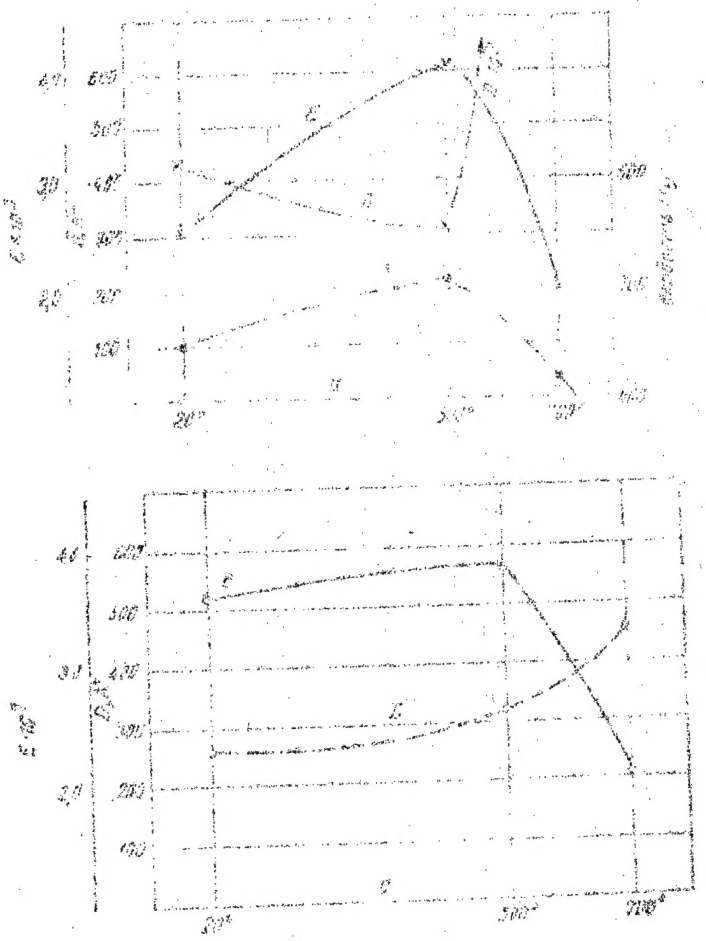
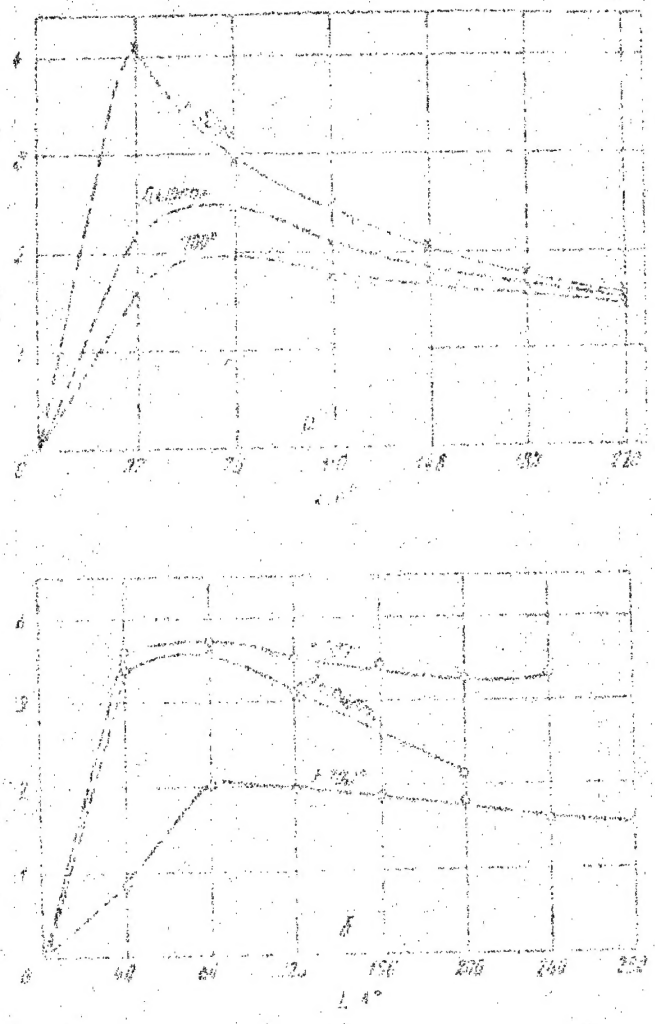


Figure 4. Microstructures and electron diffraction pictures after cold deformation (a), strength annealing at 500° (b) and weakening annealing at 700° (c). Magnification, 10,000 times.



Hardness, H_r

Figure 5. Changes in the size of blocks and micro-deformations as a result of annealing: a -- after compression of 30%; b -- after compression of 70%.



Deformation

Deformation

Figure 6. Changes in the distribution of micro-deformations as a result of annealing: a -- after compression of 30%; b - after compression of 70%.

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